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VIS, J.G.		
RRERA, D.W.		
AY, R.E.		
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OVER, W.S.		
LAN, P.M.		
NNI, B.J.		
RMAN, L.K.		
ALY, T.J.		
DAHL, T.		
BIG, J.G.		
CHINS, N.M.		
KSON, D.T.		
L, R.E.		
STER, A.W.		
RX, G.E.		
DONALD, M.M.		
ENNA, F.G.		
NTROSE, J.K.		
RGAN, R.V.		
TER, G.L.		
UTO, V.M.		
NG, T.L.		
DLIN, N.B.		
WARTZ, J.K.		
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SIFICATION:

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EG&G ROCKY FLATS

EG&G ROCKY FLATS, INC.
ROCKY FLATS PLANT, P.O. BOX 464, GOLDEN, COLORADO 80402-0464 • (303) 966-7000

August 26, 1994

94-RF-08959

ADMIN RECORD

Jessie M. Roberson
Acting Assistant Manager for
Environmental Restoration
DOE/RFFO

COLORADO DEPARTMENT OF PUBLIC HEALTH AND ENVIRONMENT (CDPHE) OPERABLE UNIT (OU) 10 SOIL GAS QUALITY ASSURANCE (QA) CONCERNS – SGS-459-94

Action: None required.

This correspondence is in response to the CDPHE letter dated July 13, 1994 regarding questions about data quality concerns pertaining to the Mobile Soil Gas Laboratory Analytical Methods used on the OU 10 Soil Gas Project. The following information was developed by EG&G Rocky Flats, Inc. to respond to the issues presented.

Issue No. 1
Concern that the data being generated is of unknown precision and accuracy and that continuing calibration check compound percent relative standard deviation (RSD) values have an acceptability of ± 50 percent while typical performance criteria for methods such as the Environmental Protection Agency (EPA) 8260 and 524.2 are 30 and 20 percent respectively.

Response
EG&G Rocky Flats Environmental Restoration Program Division (ERPD) believes that the data are of known precision and accuracy. All of the quality control (QC) parameters for this work are precisely detailed in the Standard Operating Procedure (SOP) (attached), and these have been carefully fulfilled for all work to date. All of the QC data required in the SOP have been generated for work completed to date. The QCs include Daily Reagent Blanks, Daily Calibration Standards, Internal Standard and Surrogates, Duplicate Analysis, etc.

The RSD requirements for calibration check compounds (CCCs) and systems performance check compounds (SPCCs) are 50 percent RSD (as opposed, for example, to 30 percent RSD for EPA Method 8260). The data quality objectives for this project were identified as EPA Level II. This data quality level is designed for analytical support of field activities using transportable equipment and field analysis. This type of data is generally specified for projects requiring fast-turnaround data of quality sufficient to make field decisions related to the placement of additional sample locations, site characterization, evaluation of remedial alternatives, engineering design, and monitoring during implementation.

The acceptable percent RSD of the CCCs used in the evaluation of an initial calibration, and the relative percent difference (RPD) of these compounds in the continuing calibration analysis were set at ± 50 percent, in accordance with the goal of Level II data quality. This percent RPD was selected to allow for fewer reruns under field conditions requiring rapid turnaround of large numbers of samples, and to allow for variations resulting from trap/desorption procedures compared to Methods 8260 and 524.2.

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The QC criteria employed for these soil gas analyses is more stringent than the minimum requirements of Level II data. The mobile laboratory is using a mass spectrometer (MS) for detection instead of a photoionization detector/electron capture detector employed in a similar, alternative Rocky Flats Soil Gas Method. The MS detector provides positive identification of the target analytes and better compound resolution capability than other detectors. Therefore, there is a much higher level of assurance that compounds reported are correctly identified. The MS is tuned each day to ensure that the correct ions are identified. Internal standards are introduced into each sample to adjust the sample response for individual sample introduction variations. Surrogate standards are introduced into each sample tube to detect variations in sample desorption. Initial three point calibration and a daily single point calibration are used for the calculation of the compound response factors used in the quantification of sample analytes.

Issue No. 2
Concern that the use of a ± 50 percent RSD value for initial and continuing calibration compounds indicates that the control of the method is dubious and that the odds are 50/50 that low concentration compounds will not be detected.

Response
The acceptance criteria for the initial and continuing calibration of ± 50 percent was set in accordance with the data quality objectives of the study. All of the actual CCC and SPCC data are being produced and provided with the soil gas results. For most of the calibration runs, most of the calibration compounds do have RSD values significantly less than 50 percent, and none exceed 50 percent, indicating that control of the analysis is well established with defined parameters.

The detection limits for compounds targeted in this soil gas study (defined at the 99 percent confidence level by 40 the Code of Federal Regulations (CFR) 136 Appendix B) are far below the 1 microgram per liter (µg/l) required in the work plan. A complete Method Detection Limit (MDL) study according to 40 CFR 136 Appendix B showed almost all compounds had 99 percent confidence level detection limits calculated from seven replicate analyses in the range of .01 to .03 µg/l as determined by signal to noise ratio, and the method detection limit was shown to be far below .05 µg/l by signal to noise ratio. Level II data quality does not require 99 percent confidence level detection limit according to 40 CFR 136 Appendix B.

If there is a data quality objective for which a higher confidence level is required than the QA/QC that the SOP specifies, individual data on individual compounds can be qualified from the data package being supplied.

We believe our control of the soil gas analysis for this project has met or exceeded the detailed control requirements in the SOP, and meets the data quality objectives of soil gas studies. Any calibration and individual sample analysis which has not met the project QC specifications were reanalyzed.

Issue No. 3
Request for a Method Development and Validation "report," detection limit study and methodology, quality assurance process and schedule.J. M. Roberson

Response
ERPD believes that the performance characteristics of the method, including the QC, method detection limit in accordance with Level II requirements, and quality assurance process, are fully defined in the information we have provided. If a formal report for peer review is needed, we can assist you in this.

If you should have any questions regarding this matter or require additional information, please contact Gregg A. Anderson of my staff at extension 8504.

ALP signature for SGS
S. G. Stiger, Director
Environmental Restoration Program Division

GAA:alk

Orig. and 1 cc – J. M. Roberson

Attachment:
As Stated

cc:
M. N. Silverman - DOE/RFFO
L. W. Smith - " "

WALSH STANDARD OPERATING PROCEDURES FOR OBTAINING SOIL GAS AND SOIL SAMPLES

1.0 SCOPE

Standard operating procedures (SOPs) presented in this document are intended to guide soil gas and soil sampling activities at the Rocky Flats Plant (RFP). The procedures are specifically intended to guide activities conducted by Walsh Environmental Scientists and Engineers, Inc. (WALSH) under the soil gas task of the integrated operable unit remediation investigation being conducted by Jacobs Engineering Group (Jacobs). The procedures are intended to be consistent with existing RFP Environmental Management Division (EMD) SOPs and supporting RFP EMD SOPs are referenced throughout.

2.0 GENERAL INFORMATION - SOIL GAS SAMPLING

The effectiveness of soil gas surveys depends on site-specific variables including the type of contamination, the nature of the soils, and the hydrogeologic conditions. Detection of contamination in soil vapors depends on the ability of vapors to permeate soil pore spaces and the interaction of the vapors with the soil. Compounds having low molecular weight, high vapor pressure, and low solubility diffuse more readily through vadose-zone soils.

WALSH soil gas sampling procedures will be tailored to meet project objectives. WALSH proposes using a dynamic soil gas sampling procedure involving extraction of a volume of soil gas by vacuum through hollow steel probes driven into the ground. A Geoprobe™ Model 8-M truck-mounted hydraulic ram drives probes to prescribed sampling depth. The weight of the truck (Ford F-350) and the hydraulic ram can be applied when driving the rods, enabling the probe to be driven to depths over 60 feet under suitable conditions.

2.1 Specific Equipment

- Geoprobe™ Model 8-M hydraulic ram mounted on Ford F-350 truck
- Hollow steel probes with retractable drive tips
- Steel rod for extending drive tip
- Vacuum pump with gauge for purging
- Probe bits for penetrating asphalt and concrete
- Adaptor for soil gas probe with appropriate valves and tubing
- Adsorbent tubes (Supelco Carbotrap 300™ or equivalent)
- Calibrated sampling pump

2.2 Procedure

1. Assemble soil gas sampling equipment, turn on vacuum pump making certain that the extendable tip remains open, and record the maximum vacuum gauge reading. This is the vacuum correction valve.
2. Clear the area to be sampled for utilities, cables, pipes, etc. Clear the surface area to be sampled of grass, leaves, and debris.
3. Using the Geoprobe™, drive a clean probe into the ground to the depth prescribed by Jacobs. The standard depth will be 5 feet below surface level (bsl) and variances will be specifically noted by Jacobs. If refusal is encountered at greater than 3 feet bsl, then the refusal depth will be the sampling depth. If refusal is encountered at less than 3 feet bsl, an offset drive will be attempted within 1 foot from the initial location. If refusal is encountered at less than 3 feet bsl in the offset, the location will be abandoned without sampling.
4. Retract the rods approximately 2 inches from the sample depth to open the extendable tip at the bottom of the probe. Insert a clean, 0.25-inch diameter steel rod down the center of the probe to ensure that the tip is extended and withdraw the rod.
5. Attach the adaptor with tubing and sampling manifold to the top of the probe and to the low-pressure side of the vacuum pump.
6. Run the vacuum for a time sufficient to draw a volume of gas greater than three times the volume of the sampling rods and hoses to insure that the gas sampled is truly representative of the in-situ soil gas.
   a. Monitor the flow rate with the in-line flow meter. Using the measured flow rate and a stop watch, calculate the volume of gas extracted from the soil. Record the purge volume and the cumulative time of evacuation.
   b. The corrected vacuum is calculated by subtracting the vacuum correction value from the measured vacuum. The corrected vacuum is the actual vacuum in the tubing upstream from the sample tube.
   c. Samples are considered suspect when corrected vacuum reaches more than 12 inches mercury during purge (indicating a very tight or water-saturated soil). When such excessive pressures are reached during the purge, the in-line valve will be closed and the formation will be allowed to equilibrate for 3 minutes prior to sampling.
7. Following the purge, close the in-line valve above the sampling port. Collect a sample with an adsorbent tube installed in the vacuum line connected to the calibrated flow meter pump. The sampling flow rate will fall within the working range specified by the adsorbent tube manufacturer (from 50 milliliters (ml)/minute to 100 ml/minute for Supelco Carbotrap 300™) and the total collection volume will not exceed manufacturer-specified breakthrough volumes for any of the target analytes. The collection volume will be large enough to meet reporting limits required by Jacobs. Sample tubes will be tightly capped

immediately following sample collection. Additional samples may be collected from the same location using sorbent tubes, gas-tight syringes, and/or other devices to allow for laboratory dilutions and/or duplicate analyses.

8. Measure total organic vapors through the sampling manifold using a photo-ionization detector (PID) and record for correlation with mobile laboratory data.
9. Field notes will include time of sample collection, pressure reading of vacuum at the time of sampling, sampling flow rate(s), and the amount of time over which the sample was collected.
10. Store samples in cooler for transport to WALSH mobile laboratory. Samples will be stored and transported appropriately to meet applicable holding times.
11. Remove probe from the ground. Backfill hole with native soil or a soil/bentonite mixture. Record location of sampling hole on a soil gas survey map.
12. Decontamination procedures will conform to applicable RFP EMD SOPs including FO.03, General Equipment Decontamination; and FO.04, Heavy Equipment Decontamin-ation. Reusable sampling equipment (e.g., steel rods) will be decontaminated prior to first use and following each use at the RFP.

2.3 Method Variances

Sample locations not accessible with the Geoprobe™ will be sampled by hand. Variations to the above stated method include driving rods by hand and vacuum purging with a hand pump. The hand pump will be fitted with an equivalent in-line sample port and valve.

3.0 SOIL SAMPLING

A 3-inch diameter saw will be used to core through the existing surface. If asphalt or concrete cover are present, soil samples are obtained from beneath any base course present below the surface cover. The natural soils below the base course will be sampled using a 2-inch diameter by 24-inch long stainless-steel lined California core barrel. The core barrel will be driven hydraulically using the Geoprobe™ Model 8-M hydraulic ram.

3.1 Specified Equipment

- Geoprobe™ 8-M hydraulic ram-mounted on Ford F-350 truck
- Sufficient probes so field decontamination is not required more often than once per day
- Three-inch coring saw with bit
- Deionized water for coring operations
- Two Geoprobe™-AW rod conversion kits
- Two 2- by 24-inch California barrel samplers

- Stainless-steel sample sleeves and caps

3.2 Procedure

1. All stainless-steel sample sleeves should be steam cleaned prior to use to ensure any residual cutting oil from the manufacturing process is removed.
2. Clear the area to be sampled for utilities, cables, pipes, etc. Clear the surface area as required.
3. If concrete or asphalt is present, set up the coring saw and core through existing asphalt or concrete using 3-inch bit. Use deionized water for lubrication and cooling of bit. Remove the asphalt or concrete core from the core hole.
4. Drive California core barrel equipped with a stainless-steel sample sleeve to the specified depth using the Geoprobe™ 8-M.
5. Penetration of any base course present beneath the surface concrete or asphalt may be necessary to ensure that the sample is representative of the natural soils. If the core barrel cannot be advanced due to gravels, retract the core barrel and attempt to advance the hole past the gravels with a hand auger, then reintroduce the core barrel and attempt to advance it.
6. Retract the core barrel and remove from probe rods. Disassemble the core barrel, keeping the soil sample intact in the sample sleeve. Cover both ends of the sleeve with aluminum foil or teflon tape and then cap them. Label the sample sleeve at both ends and maintain at 4° Celsius.
7. Field decontaminate the California core barrel sampler by washing with brushes and a Liquinox™ solution following by a triple rinse with distilled water. Decontamination procedures will conform to RFP EMD SOPs FO.03 and FO.04. All visible soil particles must be removed.
8. Obtain a soil gas sample in the specified manner, if required.

VOLATILE ORGANIC COMPOUND ANALYSIS

OF AIR AND SOILGAS SAMPLES BY THERMAL DESORPTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY

11.0 REFERENCES

1.1 Test Methods for Evaluating Solid Waste, SW-846, third edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Method Numbers 5030, 8000, and 8260.

1.2 Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88/039, Revised July, 1991: Method 524.2.

1.3 Compendium of Methods for the Determination of Organic Compounds in Ambient Air, EPA/600/4-89/017, June, 1988: Methods TO-1, and TO-2.

2.0 SCOPE AND APPLICATION

This method is used for the detection and quantification of organic compounds collected in air sampling tubes, particularly vadose zone soil-gas samples. This SOP serves as a guideline for the analyst but does not take the place of a proper training program.

3.0 SUMMARY OF METHOD

Measured volumes of air or soil gas samples are and passed through adsorbent traps where the volatile analytes are deposited. Each trap is sealed and delivered to the laboratory. This trap is heated and back-flushed with carrier gas to desorb the analytes and carry them onto a cryogenically cooled trap. The cryo-trap is then heated and the analytes are driven onto a chromatographic column. These analytes are subsequently detected by a mass selective detector. Compounds are identified by their mass spectra and comparison of relative retention times with those of a calibration standard. They are quantified using relative response ratios from a calibration curve. Analytes quantified at or above the statistically determined detection limits.

4.0 SAMPLE HANDLING, PRESERVATION, AND REPORTING

4.1 Samples are collected on Carbotrap 300 traps and sealed within glass tubes which are provided. Refer to the WALSH Soil Gas Sampling Procedure for further details.

4.2 Samples must be analyzed within 14 days of collection.

4.3 Samples from the Rocky Flats Soil gas survey in conjunction with Jacobs

Engineering Group will be analyzed within 4 days of collection.

4.4 Analytical data will be reported to Jacobs Engineering Group by both electronic means (computer diskette) and hard copy on a twice a week basis.

5.0 SAFETY

5.1 Many of the analytes are known carcinogens. Precautions should be taken with all samples and standards such that they are considered carcinogenic.

5.2 Solvent resistant gloves and safety glasses are required when handling carbon trap samples and standards.

5.3 Standards and solvents must be handled only in an operable fume hood.

5.4 Avoid contact with solvents and standards.

5.5 Refer to the WALSH Health and Safety Plan for further details.

6.0 INTERFERENCES

6.1 Airborne contamination in the form of extraction solvents present a major problem. A minimum of one reagent blank should be run during the course of a daily sequence, and all interfering compounds be quantified.

6.2 Carry-over contamination of the thermal desorber and gas chromatographic systems present a more controllable problem than airborne contamination, especially when samples with any individual compounds higher than approximately 3μg/L. Thorough bakeout of thermal desorber and sample tubes will alleviate this problem. If encountered, these high level samples should be immediately followed by a blank.

7.0 APPARATUS AND EQUIPMENT

VOLATILE ORGANIC COMPOUND ANALYSIS
OF AIR AND SOILGAS SAMPLES BY THERMAL DESORPTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY

7.1 Sample Preparation

7.1.1 Gas Tight Syringes - Assorted sizes.

7.1.2 Static Dilution Flasks - 1.0L.

7.1.3 Adsorbent Traps - Carbotrap 300 or equivalent.

7.2 Thermal Desorption System - System consisting of thermal desorption device, cryogenic trap, and heater. Designed to accept 7 inch x 0.25 inch sample tubes.

- Tekmar Aerotrap 6000 or equivalent.

7.3 Gas Chromatograph

7.3.1 Analytical system complete with gas chromatograph suitable for thermal desorption sample introduction.

- HP-5890 or equivalent.

7.3.2 Capillary Column (equivalents acceptable)

- DB-VRX; J&W Scientific; 0.33mm x 60m, 1.8μ film thickness.

7.3.3 Detector

- Mass Spectrometer - HP 5972 or equivalent.

7.4 Data Collection - Data system capable of acquiring and storing raw data from the mass spectrometer.

- HP Vectra 486/66U or equivalent.

7.5 Data Reduction - Software capable of reducing raw data into hard copy and electronic media for reporting to the Jacobs Engineering Group.

- HP Enviroquant Software for GC/MS or equivalent.

7.6 Miscellaneous

7.6.1 Drying Oven.

7.6.2 Freezer.

7.6.3 Refrigerator.

7.6.4 Analytical Balance.

7.6.5 Ultra High Purity Helium.

7.6.6 Liquid Nitrogen.

8.0 REAGENTS

8.1 Methanol (Purge and Trap Grade Methanol)

9.0 STANDARDS

9.1 All standards must be less than 2 years old. Stock standards and unopened standards in methanol are stored in the freezer at less than 0° C. Working standards should be made up as needed.

9.2 Initial and Daily Calibration

9.2.1 502.2/8260 Analyte List (equivalent acceptable)

- Restek VOACYL III - 2.5μg/ml in nitrogen.

- Restek Custom 502.2 Calibration Mixes #2-6 - 20,000μg/ml in methanol.

9.3 Internal Standard (equivalent acceptable)

9.3.1 Restek Custom 8260 Internal Standard Mix - 20,000μg/mL in methanol.

9.4 Surrogate Standard (equivalent acceptable)

9.4.1 Restek Custom 8260 Surrogate Standard Mix - 20,000μg/mL in methanol.

9.5 Laboratory Control Standard (LCS)/Matrix Spiking Solution (equivalent acceptable)

- Restek Custom VOA Matrix Spike Mix - 20,000μg/mL in methanol.

9.6 Standards Preparation: Working standards are prepared by injecting high concentration standard solutions #2 - #6 into a precleaned 1L static dilution bottle with glass beads. The contents of the VOACYL III may be injected directly onto the adsorbent trap. Prior to instrument calibration the dilution bottle should be heated to 65°C and shaken to evaporate all liquids.

10.0 PROCEDURE

10.1 Preparation

10.1.1 Thermal desorption tubes must be conditioned at 375°C for a minimum of 8 hours prior to use.

10.1.2 Liquid Nitrogen must be available for cryo trap.

10.2 Instrument Conditions

10.2.1 Typical Aerotrap Conditions

- Tekmar Aerotrap 6000

Sample Sweep Time:	4 minutes
Sample Sweep Condition:	Pre-Cool
Sample Desorb Temperature:	30°C
Sample Desorb Time:	8 minutes
Sample Tube Bake Temperature:	375°C
Sample Tube Bake Time:	8 minutes
Cryo Trap Temp:	-190°C

Trap Desorb Temperature:	240°C
Trap Desorb Time:	4 minutes
Trap Desorb Preheat:	200°C
Trap Bake Temperature:	260°C
Bake Time:	6 minutes
Cryo Trap:	#19-Glass Beads

10.2.2 Typical Gas Chromatograph Conditions

- DB-VRX Column

Carrier:	Helium
Electronic Pressure Control Conditions:	Varied (See Below)
Initial Temperature:	40° C
Initial Time:	8 minutes
Oven Program Rate:	8° C/minute
Final Temperature:	230° C
Final Time:	2 minutes
Total Run Time:	32.75 minutes

- Electronic Pressure Control Conditions

Initial Pressure:	12.6 PSI
Initial Time:	0 minutes
Pressure Program Rate 1:	99 PSI/min
Pressure 2:	50 PSI
Time 2:	0.25 minutes
Pressure Program Rate 2:	99 PSI/min
Pressure 3:	12.6 PSI
Time 3:	7.04 minutes
Pressure Program Rate 3:	0.57 PSI/min
Pressure 4:	26.2 PSI
Time 4:	0.89 minutes

10.2.3 Typical Detector Operating Conditions

- Mass Spectrometer: HP 5972
  Acquisition Mode: Scan (2.2 scans/second)
  Mass Range: 35-350 AMU
  Tuning Criteria:
  Tune Evaluation Standard: 4-Bromofluorobenzene
  Mass Intensities:

Mass 50:	15 to 40% of mass 95
Mass 75:	30 to 60% of mass 95
Mass 95:	base peak
Mass 96:	5 to 9% of mass 95
Mass 173:	< 2% of mass 174
Mass 174:	> 50% of mass 95
Mass 175:	5 to 9% of mass 174
Mass 176:	95 to 101% of mass 174
Mass 177:	5 to 9% of mass 176

10.3 Daily Quality Control Preparation

10.3.1 Daily Calibration Standard: A standard verification of the initial calibration e must criteria for SPC and CC Compounds.

- From a known concentration Static Dilution Bottle which has been heated to 65°C draw a known amount of air standard.
- Inject directly into clean air sampling tube.
- From a second Static Dilution Bottle which has been kept under the same conditions, draw a known amount of Internal/Surrogate standard mix and inject onto the same trap.
- From a VOACYL III stored at room temperature draw an aliquot of the gases and inject onto the trap.

- Desorb and analyze trap contents for instrument calibration.

10.3.2 Daily Reagent Blank: Blank samples used to quantify system contamination. A minimum of one reagent blank should be run in the course of a daily sequence. Blanks should contain less than the reporting limit for all analytes with the exception of common laboratory contaminants (methylene chloride, acetone, toluene).

- Draw 250μL of IS/SS into a Gastight syringe from a heated Static Dilution Bottle.
- Connect clean, conditioned sample tube to air pump and begin flow.
- Transfer IS/SS into air sampling tube.
- Desorb and analyze trap.

10.3.3 Laboratory Control Standard (LCS): Standard which contains the parameters of interest is used to judge overall system performance and stability. Only used for EPA level 3 - 5 data.

- Draw 250μL of IS/SS and 250μL of LCS into Gastight syringes from heated Static Dilution Bottles.
- Connect clean, conditioned sample tube to air pump and begin flow.
- Transfer IS/SS and LCS into air sampling tube.
- Repeat for LCS 2.

10.3.4 Matrix Spike: Sample which is spiked with the LCS solution is run to show any matrix interference problems that may exist with a particular sample. An MS should be performed once for every 20 samples and for every matrix present in a sample set. Matrix spikes are run only when EPA level 3 - 5 data is specified.

- Draw 250μL of IS/SS and 250μL of LCS into Gastight syringes from heated Static Dilution Bottles.
- Connect sample tube containing a sample to air pump and begin flow.
- Transfer IS/SS and LCS into air sampling tube.

10.4 Sample Preparations:

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- Draw 250μL of IS/SS into a Gastight syringe from a heated Static Dilution Bottle.
- Connect sample tube containing a sample to air pump and begin flow.
- Transfer IS/SS into air sampling tube.

11.0 CALIBRATION

11.1 A three point calibration curve is used to determine the linear range of the GC/MS for EPA levels 1 and 2. A five point curve is used when EPA levels 3 - 5 are required. The calibration curve determines the working range of the instrument thus the low standard should be within 5 - 10 times the instrument detection limit. Response factors (RFs) and retention times are tabulated using computer data system.

- Typical Calibration Analyte amounts:

  - 0.100μg, 0.500μg, and 2.00μg for a 3 point curve.
  - 0.100μg, 0.200μg, 0.500μg, 1.00μg, and 2.00μg for a 5 point curve.

11.2 Calibration Acceptance Criteria

11.2.1 Using data collection software, tabulate RFs and retention times. Calculate linear fits to the calibration curve.

11.2.2 All parameters must meet the quality control criteria listed in the quality control section.

12.0 CALCULATIONS

12.1 Relative Response Factors (RRF):

$$RRF = \frac{(\text{Conc. Int. Standard } [\mu g/l])\ (\text{Area Analyte})}{}$$

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(Area Int. Standard) (Conc. Analyte [μg/l])

12.2 Analyte Concentrations:

$$\text{Conc.} = \frac{\text{(Area Analyte) (Conc. Int. Standard)}}{\text{(Area Int. Standard) (RRF) (Dilution Factor)}}$$

13.0 QUALITY CONTROL

13.1 Daily reagent blanks are run to verify the cleanliness of the system. Blanks should contain less than the reporting limit for all analytes with the exception of common laboratory contaminants (methylene chloride, acetone, toluene).

13.1.1 If blanks contain analytes other than common laboratory contaminants at a concentration greater than the reporting limit, they must be re-prepared and re-analyzed to meet the requirements.

13.1.2 If blanks contain common laboratory contaminants at a concentration greater than the reporting limit, the reporting limit will be raised by two times the blank concentration.

13.2 The initial calibration must have a minimum of five points for each analyte if EPA levels 3, 4, or 5 are required, or three points if EPA levels 1 or 2 are necessary. The calibration curve should define the working calibration range. The maximum percent relative standard deviation of the curve for CCCs must not exceed 50%. The minimum relative response factor for SPCCs must not be lower than 0.200 (0.150 for Bromoform).

If all requirements for the initial calibration are not met, a new initial calibration must be run until all requirements are met.

13.3 A daily calibration standard is run to verify the initial calibration curve every 12 hours of instrument operation. SPCCs must have a minimum RRF of 0.200 (0.150 for Bromoform) and CCCs must have a maximum %RPD of 50%.

13.3.1 If any analyte is out of the acceptable recovery range, the calibration standard must first be re analyzed.

13.3.2 If any analyte is consistently out of the acceptable recovery range, corrective action should be taken to determine the root of the problem and a new initial calibration curve should be run and the daily calibration standard should be re analyzed.

13.4 For EPA levels 3 - 5 an LCS containing the parameter of interest should be run for every 20 samples analyzed. Use laboratory generated control limits to initiate corrective actions.

$$RPD = \frac{[(\text{Initial Sample Result} - \text{Duplicate Sample Result})*(100)]}{\text{Average of the Two Results}}$$

13.4.1 If the LCS has any analyte out of the acceptable recovery range, it must be re-analyzed.

13.4.2 If any analyte is consistently out of the acceptable recovery range, a new stock solution of LCS should be opened and analyzed. If any analyte remains out of the acceptable recovery ranges, a new initial calibration should be run and the LCS must be re-analyzed.

13.5 For EPA levels 3 - 5 one matrix spike and duplicate must be run for every 20 samples analyzed. Recoveries should fall within laboratory defined control limits.

13.5.1 When the analyte concentration is three times the spike concentration or greater, the spike recovery is not reported because it exceeds the calibration range.

13.5.2 Matrix spike recoveries which are out of the acceptable range are evaluated by the analyst and noted in the raw data file. No actions is taken unless determined otherwise by an experienced analyst.

13.6 Internal standard recoveries for all samples and spikes should fall within -50% to +100% of the daily continuing calibration.

13.6.1 If a sample internal standard recovery is out of the acceptable recovery limits, the sample should be re-analyzed.

13.6.2 If a sample is re-analyzed and the internal standard recovery is still out of the acceptable recovery range, the data should be reported and matrix interference should be noted in the raw data file.

13.7 Surrogate standard recoveries for all samples and spikes should fall within 40% to 180% of the daily continuing calibration.

13.7.1 If a sample surrogate standard recovery is out of the acceptable recovery limits, the sample should be re-analyzed.

13.7.2 If a sample is re-analyzed and the surrogate recovery is still out of the acceptable recovery range, the data should be reported and matrix interference should be noted in the raw data file.

13.8 Samples must be analyzed within the 14 day holding time which begins on the date of sampling.

13.8.1 If holding times are exceeded, results are considered unusable for regulatory work.

13.8.2 For non-regulatory work, detection limits and positive results are considered estimated values and the holding time exceedance is noted in the raw data file.

13.9 The laboratory established retention times should be used as a guide for identification of analytes. Analytes should fall within +/- 0.05 of the daily continuing calibration standard relative retention times. Internal standards should fall within 0.5 minutes of the retention times in the continuing calibration standard.

13.9.1 If an analyte falls outside of this retention time window, a qualified analyst should determine if the analyte should be reported.

13.9.2 Analyte retention times are updated daily with the daily calibration standard and throughout the run using the internal standards as reference peaks.

13.10 Refer to the most recent detection limit study for analyte reporting limits.

13.11 For EPA level 3 - 5 data trip blanks should be prepared by conditioning and

sealing a thermal desorption trap in the lab and have it accompany a set of traps out into the field. Upon it's return the trip blank should be analyzed to determine whether or not there exists any cross-contamination.

Table I

System Performance Check Compounds

(SPCC)

Chloromethane

1,1-Dichloroethane

Chlorobenzene

Bromoform

Bromobenzene

Continuing Calibration Check Compounds

(CCC)

Vinyl Chloride

1,1-Dichloroethene

1,2-Dichloropropene

Toluene

Ethyl Benzene